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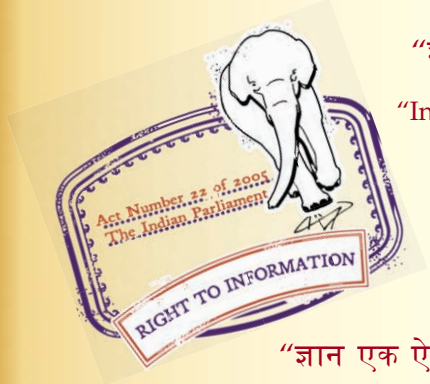

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METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 1 DETERMINATION OF COPPER AND LEAD BY ELECTROLYTIC METHOD

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 1 DETERMINATION OF COPPER AND LEAD BY ELECTROLYTIC METHOD

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 1 DETERMINATION OF COPPER AND LEAD BY ELECTROLYTIC METHOD

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 1) (First Revision) was adopted by the Bureau of Indian Sandards on 22 July 1987, after the draft finalized by the Methods of Chemical analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 4027, first published in 1967, covered determination of copper, lead, tin, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc and antimony in bronzes. While reviewing this standard, the Sectional Committee decided that it is convenient to revise this standard in series of parts which, on publication will supersede the relevant method for determination given in IS : 4027-1967*. This part is one of that series and covers the determination of copper and lead by electrolytic method. The other parts are as follows:

Part 2 Determination of manganese by photometric method

Part 3 Determination of phosphorus by volumetric method

Part 4 Determination of nickel by photometric method

Part 5 Determination of tin by iodimetric method

Part 6 Determination of zinc by complexometric (EDTA) method

Methods for chemical analysis of other constituents in bronzes, namely, aluminium, iron, silicon and antimony are under preparation and will be published in subsequent parts of above series.

0.3 In this revision, determination of lead by the gravimetric and colorimetric methods, stipulated in earlier edition, have been deleted. The figures describing the details of electrodes are also deleted.

*Methods of chemical analysis of bronzes.

0.4 The methods of analysis prescribed in this standard may primarily serve as referee methods and may also be used by the laboratories for their day-to-day work. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 1) prescribes a method for determination of copper and lead in the ranges as specified in the relevant Indian Standards on bronzes.

NOTE — The method is not applicable when manganese is present in bronzes and also when lead is of the order of 0·1 percent.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS : 1817-1961†.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070-1977‡) shall be employed in the test.

4. DETERMINATION OF COPPER AND LEAD BY THE ELECTROLYTIC METHOD

4.1 Outline of the Method — The sample is dissolved in nitric acid, and copper and lead are electrolytically deposited and weighed.

NOTE — This method is not applicable for too low as well as higher content of lead.

4.2 Apparatus

4.2.1 *Electrodes* — The following platinum electrodes are recommended, but strict adherence to the shape and size of the electrodes is not essential. For agitation of electrolyte in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed.

*Rules for rounding off numerical values (*revised*).

†Methods of sampling non-ferrous metals for chemical analysis.

‡Specification of water for general laboratory use (*second revision*).

4.2.2 *Cathode* — It may be formed either from plain or from perforated sheet or from wire gauge.

4.2.2.1 Gauge cathodes preferably made from gauge containing 400 meshes/cm^2 should be used. The wire used for making gauge should be approximately 0·20 mm in diameter. Cathodes should be stiffened by doubling the gauge for about three millimetres on the top and the bottom or by reinforcing the gauge at the top and bottom with platinum ring or band.

4.2.2.2 The diameter of the cylinder should be approximately 30 mm and the height 50 mm. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1·5 mm. It should be flattened and welded throughout the entire length of the gauge. The overall height of the cathode should be approximately 130 mm.

4.2.3 *Anode* — When the amount of lead in the sample is less than 4·0 mg, a sprial anode should be used. It should be made from 1·0 mm or larger platinum wire formed into sprial of seven turns with a height of approximately 50 mm and diameter of 12 mm, the overall height being 130 mm.

4.2.3.1 When the amount of lead in the sample is more than 4·0 mg, the gauge anode should be used. It should be made of the same material and of the same general design as platinum gauge cathode mentioned under **4.2.2**. It should be approximately 12 mm in diameter and 50 mm in height, the overall height being 130 mm.

4.3 Reagents

4.3.1 *Dilute Nitric Acid* — 1:1 (*v/v*).

4.3.2 *Hydrobromic Acid* — 48 percent.

4.3.3 *Urea* — Solid.

4.3.4 *Sulphamic Acid* — Solid.

4.3.5 *Hydrogen Sulphide Solution* — Saturate dilute sulphuric acid (1:99) with hydrogen sulphide gas. Prepare fresh as needed.

4.3.6 *Ethanol* — 95 percent (*v/v*).

4.4 Procedure

4.4.1 Weigh 2·500 g of sample, dissolve in 25 ml of dilute nitric acid and evaporate to syrupy consistancy. Add 50 ml of hot water and allow to stand on a steam bath for one hour. If there is any

opalescence or precipitate, add paper pulp, filter and wash several times with acidulated water. Reserve the filtrate.

4.4.1.1 Transfer the residue to silica crucible and ignite. Add 10 to 12 ml of hydrobromic acid, evaporate to dryness and ignite. Take up residue with dilute nitric acid and boil to expel brown fumes. Add to the filtrate reserved under **4.4.1**. Repeat the hydrobromic acid treatment till tin is completely volatilized.

4.4.1.2 Adjust the volume of the solution to 150 ml. Add 0·5 g of urea or 0·1 g of sulphamic acid and boil for a few minutes. Insert the tared electrodes, cover, the beaker with split watch-glasses. Electrolyse with a current of 5 A/dm^2, with constant stirring. When the solution is colourless, wash down the cover glasses, electrodes and sides of the beaker, raising the level of liquid slightly. Continue passing the current noting whether or not copper is being plated on the newly exposed surface of platinum cathode. If no copper appears, transfer about one millilitre of the solution to a spot plate and test for copper with a few drops of freshly prepared acidified hydrogen sulphide solution.

4.4.1.3 As soon as the deposition is complete, lower the beaker slowly while washing the cathode with water without stopping the current. Remove the cathode, rinse it with water and then dip in two successive baths of ethanol. Dry for three to five minutes in an oven at 105°C, cool and weigh the deposit immediately as metallic copper. Remove the anode, rinse thoroughly with water, and dry the anode in an oven at 100°C for 30 minutes. The deposit, being fragile, should be handled with care. Cool the anode and weigh as lead peroxide.

4.5 Calculation

$$\text{Copper, percent} = \frac{A}{C} \times 100$$

$$\text{Lead, percent} = \frac{B \times 86{\cdot}62}{C}$$

where

A = mass in g of the copper deposit,

B = mass in g of the lead peroxide deposit, and

C = mass in g of the sample taken.

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